Technosols designed for rehabilitation of mining activities using mine spoils and biosolids. Ion mobility and correlations

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Abstract

The restoration technologies in areas degraded by extractive activities are more efficient under the use of their own spoils. Reducing deficiencies in physical properties, organic matter, and nutrients with a contribution of treated sewage sludge is proposed. This experiment was based on a controlled study using columns. The work was done with two limestone quarry spoils, both very rich in calcite. Two biosolids doses were undertaken (30,000 and 90,000 kg/ha of sewage sludge) in addition to a different quarry spoils used as substrates. The water contribution was provided by a device simulating short duration rain. The leached water was collected 24 hours after the last application. Nitrate, ammonium, phosphate, sulfate, and chloride ions were determined, as well as the pH and electrical conductivity. The electrical conductivity limit value is <1000 µS/cm. These values will be met from the fourth irrigation application onward, while the values up to that point were far superior. Significant nitrate concentrations appeared that may pose an environmental contamination risk. A comparison between the concentrations of the chemical elements obtained in the leachates from our experiment and the established limit values for water of the third quality group has been performed. The electrical conductivity correlated well with the cations, with the exception of potassium. For sulfates, significant correlations were obtained with the Mg^{2+}, Ca^{2+}, and K^+ cations. The chlorides showed excellent correlation with the sodium.

Keywords Ion mobility • Irrigation • Quarry spoils • Sewage sludge

1 Introduction

The restoration of extensive areas degraded by mining activities requires the use or their own waste materials (Jordán et al. 1998; Tedesco et al. 1999; Ram et al. 2006; Jordán el al. 2006; Almendro-Candel et al. 2014, Galán et al. 2014). These materials do not possess the necessary fertility to ensure a successful process of restoration (implementation of adequate plant cover). Therefore, it requires the addition of organic amendments to achieve efficient substrate (Jordan et al. 2008). The obligation to restore abandoned quarries and the correct application of biosolids is guaranteed by the legislation on waste management, biosolids and soil conservation (Jordán et al. 2008). Technosols are one of the latest additions to the World Reference Base for Soil Resources (FAO 2006). This new reference soil group contains a large range of artifacts and materials of natural and anthropic origin. They include a variety of refuse-based soil-like quarry spoils, landfills, ashes, or sludges, whose properties and pedogenesis are dominated by their technical origin (FAO 2006). An adequate Technosol selection, based on its nature and intrinsic properties, can constitute a valuable and cost-effective solution for soil remediation and waste management (Novo et al 2013). Sewage sludge application in restoration has demonstrated its efficiency in previous studies (Clapp et al. 1986; Albiach et al. 2001; Pond et al. 2005; Jordán et al. 2008; Soriano-Disla et al. 2014). The use of treated sewage sludge can be a guarantee of success in the restoration of areas affected by mining activities, but it is important to preserve the conservation of the environment with less risk of contamination of surface and groundwater. Many physical and chemical properties in soils amended with sludge, such as water retention capacity, aggregate stability, contribution of N, P, and other nutrients to crop growth, depend, to some extent, upon the quantity of organic matter in the sludge that is added (Roldán et al. 1994). Knowledge about the quantity of organic matter in the sludge can be used to estimate the quantities that must be applied to the soil (Giovannini et al. 1985). Use sewage sludge and quarry spoils to construct Technosols represent an innovative strategy of waste management, whose application allowed the species to grow and develop (Novo et al. 2013). The materials resulting from the acquisition of arid particles produced from crushed limestone present very limited results in restoration because of their chemical and mineralogical characteristics. The materials resulting from limestone extraction, with elevated soil stoniness and low fertility, are usually used in restoration, and the efficiency depends upon the organic corrections (Jordán et al., 2008).

The studied area, Sierra de Callosa has an area of 8 km^2 and a maximum altitude of 566 masl (Jordán et al. 2008). Geologically, it is found to be primarily comprised of carbonated rocks. Limestones have been worked for many thousands of years, initially for building stone and agricultural lime and more recently for a wide range of construction and industrial uses. In former days, limestone quarries were small operations with limited production due to the labor-intensive nature of the business and restricted markets for stone. Stone quarries can answer the demand of aggregates for concrete applications (Jordán et al. 2008).

The main objective is to evaluate the ion mobility and correlations in columns packed with quarry spoils from a limestone quarry amended with biosolids.

2 Materials and methods

2.1 Substratum

The experiment was carried out using two different limestone quarry spoils, both very rich in calcium carbonate (450-750 g/kg). The first, of poor quality, originates from the crushed limestone (Z). It is composed of coarse materials (up to 75 % by weight) and sand. The other tested waste material comes from the extraction of limestone. This waste was formed by the levels of interspersed non-limestone materials and remains of stripped soils (D). This usually presents more balanced textures but with elevated heterometry soil stoniness (up to 60 % by weight), and is richer in clays (approx. 25 % by weight). Analytical parameters were determined in accordance with ISRIC (1993). Organic matter was quantified by wet oxidation, using the method of Walkley and Black (1974); total nitrogen by the Kjeldahl method; pH in a 1:2.5 soil/water suspension; the $CaCO_3$ equivalent with a Bernard calcimeter; by atomic absorption (Ca, Mg, Fe, Cu, Mn, Zn) and flame atomic emission spectroscopy (Na,K). Particle size distribution was determined by the pipette method.

The characteristics of the mineral substrata employed appear in Table 1. Both these materials were amended with the biosolid according to Alcañiz et al. (1997) quarry restoration methodology.

2.2 Sewage sludge

The biosolid used in this experiment comes from a wastewater treatment plant located near Aspe (Alicante). Prior to the composting process, the sludge needs to be mixed with a bulking agent, a supporting structure that favors aeration, absorbs humidity, and furthermore contributes with organic matter. Chopped hay and sawdust are used as bulking agent, and silos exist for their storage. Hay favors aeration, sawdust absorbs humidity, and both materials constitute sources for carbon. The composition by volume of the sludge-bulking agent mixture is 50 % sludge, while the remaining 50 % is 1/4 hay and 3/4 sawdust. This sludge-bulking agent mixture progresses through the composting tunnel and is simultaneously homogenized by a tumbler, which in addition to permitting the progress and homogenization of the mixture, promotes its aeration. During the first weeks, the mixture is placed upon a porous base connected to an air injection system using fans or blowers, which maintains discontinuous forced aeration. Afterwards, the aeration is passive and natural (Clapp et al., 1986; Hernández-Fernández, 1986).

For the biosolid analysis, total content of metals was determined following microwave digestion using HNO_3 and analyzed by inductively coupled plasma mass spectrometry. In the solution thus obtained, the solubilized elements except for nitrogen were assessed. This was determined by the Kjeldahl method, which quantifies the organic nitrogen and ammonium contents within the sample. The easily oxidizable organic carbon was estimated by sulfochromic digestion and subsequent assessment with Mohr's salt, while the easily oxidizable organic matter was calculated by multiplying the organic carbon by 1.72. The total organic matter was obtained by calcination in a muffle furnace at 500 °C for 2-4 h. Table 3 shows sewage sludge composition.

2.3 Columns

The experiment was based on a controlled study using columns. Fifteen columns, each 30 cm tall (Fig. 1), were constructed from 10.5 cm internal diameter PVC pipe that was cut into two 15 cm lengths. Each column was divided into two different 15 cm sections, the first one from 0 to 15 cm and the second from 15 to 30 cm. For each treatment three replicates were done (Table 1).

2.4 Treatments

Two treatments and a control were applied (30,000 kg/ha and 90,000 kg/ha), which depended upon the quantity of sludge applied and the experimental design (Table 3). The sludge was applied on the surface and mixed with the soil, simulating a plowing or tilling action, producing a homogenous mixture within the uppermost 15 cm of soil (Almendro et al., 2007; Almendro et al., 2014).

2.5 Irrigation

In order to establish the closest parallels between real conditions and those of the experiment, the soil contained in the columns was irrigated (eight applications) using tap water. The first five irrigations occurred every two weeks and the last three once per month. The irrigation applications lasted six months. Collection of the leached water was carried out 24 hours after the last application. This irrigation is equivalent to weekly rainfall of 100 mm (rainfall conditions during times of maximum abundance). The contribution of water was provided by a device that simulated short rainfall or a flood irrigation system that covered the surface and then percolated into the soil (Almendro et al., 2014). It consists of a plastic recipient with holes punched in the bottom (Fig. 1).

In order to control what was incorporated into the soil columns by the irrigation, water samples were taken from each column. Irrigation water characteristics were determined first (Table 4), i.e., pH, electrical conductivity, the Na^+, K^+, Ca^{2+}, and Mg^{2+} cations, as well as the Cl^-, SO_4^{2-}, NO_3^- and PO_4^{3-} anions (Cánovas 1980). The parameters analyzed in the leachates followed the Standard Methods (APHA Standard Methods, 2005). Na, K, Ca, Mg were determined by atomic absorption spectrometry following acid digestion in a microwave. Cl^- by titration by the Mohr method; SO_4^{2-} based on the formation of a colloidal form with barium; soluble PO_4^{3-} using the vanadomolybdophosphoric acid method.

2.6 Leachates

The electrical conductivity was determined by an electrical conductivity meter, which incorporates a conductivity cell, considering 25 ºC as the reference temperature, according to current analysis methods (APHA Standard Methods, 2005).

The chloride content was determined by the Mohr method, based on the formation of silver chloride, an insoluble salt, detecting the turning point by the appearance of a red precipitate of Ag_2CrO_4, a compound used as an indicator (APHA Standard Methods, 2005). Sulfates were determined following the Rodier (1981) nephelometric technique. The nitrate content is determined by second-derivative ultraviolet spectroscopy following the Sempere et al. (1993) methodology.

The method for the determination of phosphorous is based on the formation of a phosphomolybdic complex in an acid medium, reduced by ascorbic acid, producing a blue coloration that is measured at 825 nm. The phosphorous is measured as a phosphate ion.

The method for determining ammonium is based on the development of indophenol blue by reaction of ammonium ions treated with a solution of sodium hypochlorite and phenol in the presence of nitroprusside acting as a catalyst. The Na^+, K^+, Mg^{2+}, and Ca^{2+} ions are measured directly in the sample or in appropriate dilutions by atomic emission spectrophotometry in the case of the first two ions, and by atomic absorption for the last two.

2.7 Statistical Analysis

Statistical analysis (based in the Student's t-test at 95% and ANOVA F test) were used to determine the statistical significance of the treatments and differences between means. Simple linear regression analysis was applied to the developed experimental data. The squared correlation coefficient (R^2) represents the proportion of the variation of a variable that is explained by its linear association with another variable.

3 Results and discussion

3.1 Substratum and sewage sludge properties

The substratum used has an alkaline reaction indicating that most nutrients could manifest problems of availability (Jordán et al., 2004). In cases like this, acidifying amendments are necessary to lower the pH, facilitate element mobility, and improve the soil structure. The substratum has a relatively low nutrient content. Calcium is the element found in the greatest proportion in the waste materials. Potassium and Na concentrations (Table 1) are similar to the surrounding degraded agricultural soils (Jordán et al., 2004). This is reflected in the soil's electrical conductivity because these cations, especially Na, have a high mobility (Jordán et al., 2004; Jordán et al., 2008). The equivalent calcium carbonate content is very high, as it is typical for these types of residues. The organic matter content is very low (Table 1), just like that for available phosphorous and Kjeldahl nitrogen compared with the desired normal content for a cultivated soil.

The biosolid (Table 2) presents low contents of P and K, with medium contents for calcium and magnesium, all within the ranges cited by Juárez et al., (1987). The total Na content is of some importance, but cannot be considered dangerous for the soil (Jordán et al., 2004). Analytically control of the sludge at the time of its incorporation is important, especially with regards to Na, as this element may cause soil salinity problems and alter its structure (Moreno Sánchez et al., 1986). The C/N ratio is 12, indicating that the organic matter is partially mineralized and, therefore, the sludge can enhance soil fertility (Hernández Fernández et al., 1986; Roldán et al., 1994). The sewage sludge selected has an organic matter content that is very suitable for agricultural use (Table 2).

3.2 Leachate analysis

The leachates of the mineral substratum can serve as a point of reference for possible contamination that may appear in groundwater when sewage sludge is applied as an amendment (Jordán et al., 2008). There are some physical–chemical parameters concerning water that is destined to spill into aquatic resources. Some physical-chemical parameters concerning water that can attain the aquatic resources are regulated by the European Union Directive 76/464, transposed to the Spanish norm by the Hydraulic Public Domain Regulation, approved by Royal Decree 849/1986. The Public Administration of Water Regulation and the Hydrologic Plan, approved by RD 927/1988, states that the water quality objectives will be defined in the respective Hydrologic plans, depending upon the foreseen water uses. The values in Table 5 are solely illustrative, since in the case of leachates (washing waters) it does not deal with waters directly spilled into the aquatic resource. Furthermore, the inexistence of a limit value for nitrates calls attention.

3.2.1 pH

The pH values were found to be within the legislation limit value range (5.5-9). No significant changes in the pH were produced between treatments (7.24-8.38); an acidifying trend was only seen in the first and third sampling of the treatments. Over time, it was observed that the pH values were more similar to that of the irrigating water (7-7.8) before being added to the soil. The lowest pH values coincided with the beginning of the experiment (incorporation of residual matter and beginning of irrigation) and when the greatest degradation of the organic matter appears to have occurred, between the second and third irrigation. Similar results have been obtained by other authors using quarry spoils (Alcañiz et al., 1997; Almendro-Candel et al., 2007; Jordán et al., 2008).

3.2.2 Electrical conductivity

With respect to the control (< 800 µS/cm), an increase in electrical conductivity was observed in the water collected from the columns treated with biosolid (Table 5). This is due to the resulting washing of the soluble salts contained in the biosolid applied to the soil. The electrical conductivity values were closely related with the dose of biosolid applied. Thus, the electrical conductivity values were only worrisome during the first three irrigation applications (6700-1234 µS/cm); beginning with the fourth and particularly the fifth ones, the electrical conductivity values stabilized as the irrigations evidently washed out the salts (< 500 µS/cm). The electrical conductivity limit value is < 1000 µS/cm. These values will be met from the fourth irrigation onward, while the values up to that point were far superior. However, the quality of the aquifer's groundwater is quite poor, reaching conductivities of 5000 µS/cm, and so EC would not represent any environmental risk to the aquifer.

3.2.3 Inorganic nitrogen forms

Two of the three inorganic forms of nitrogen in the leachates are discussed: nitrates and ammonium (Table 5). The nitrites analyzed in the wash water were close to the detection limit of the technique used. Their results are not discussed because they were not significant.

An increase in nitrate concentration was observed in the water resulting from the wastes treated with sludge with respect to the control waste (Table 5). The treatments with high biosolid doses (90,000 kg/ha) were those that contributed to the higher nitrate contents to the water. The highest NO_3^- concentration in the leachates occurred in irrigations 1, 2, and 3 (2510-268 mg/L). From the fourth irrigation onward, the leaching of this anion was scarce (< 100 mg/L). The nitrates exceeded the recommended values in the two treatments. In any case, these high nitrate concentrations would drop with the restoration and development of vegetative cover, which would assimilate a large portion of the nitrates. Significant nitrate concentrations appeared that may pose an environmental contamination risk. The Code of Good Agriculture Practices does recommended the specific quantities of nitrogen to applied per hectare annually, which in the case of manure, oscillates between 170 and 210 Kg/ha. However, there is no mention of biosolid. Similar experiments carried out by Almendro Candel et al. (2007) demonstrated that this mineral column, under the conditions prevailing in this study, does not retain NO_3.

Nitrification is favored with good aeration and free drainage (Skiba and Ball, 2002). This effect increased in the combined treatment of sewage sludge compost and saline wastes. For both treatments with sewage sludge compost, nitrate leaching over time could be associated with the biological activity of the biosolids, and the evolution followed a similar trend to that observed by Santibáñez et al. (2007) in Chilean mining tailings.

The values of ammonium were only significant for the first irrigation (0.2-1.6 mg/L). This cation increased with the biosolide dose, and the differences decreased over time. The ammonium quantities were far inferior to those obtained for NO_3^-.

3.2.4 Anions

The limit values for the chlorides (< 700 mg/L), phosphates (< 27 mg/L), and sulfates

(< 800 mg/L) were very superior to those obtained in all the irrigations. A certain tendency of the phosphorus to increase with the sludge dose treatment was observed. The highest values (< 17 mg/L) were obtained in the columns filled with stripped soil (D) and with the applications equivalent to 90,000 kg/ha of composted and treated sludge. The recommended concentration limits (Table 5) were not exceeded in any treatments. High natural limestone in the wasted impeded in part the displacement and loss of the P contained in the sewage sludge, which can precipitate as calcium phosphate Albiach et al, 2001).

Chlorides and sulfates are involved in plant nutrition (Almendro et al., 2014). Furthermore, they are very relevant quality control parameters for water. Significant differences appeared in the chlorides between the treatments with biosolids and control. Probably, the most influential factor when determining the Cl^- in the leachates is the contribution from the sewage sludge, as the substrata used (Z and D) contains abundant salts. In fact, in the first two irrigations, high chloride values resulted in the control columns with the presence of limestone spoils (Z or Z+D) with lower values in the control columns filled with stripped soil (D). This observation demonstrates the contribution of chlorides by the washing of the limestone spoils (Z) used as a mineral substratum. The chlorides were completely washed out in the first three irrigations. In the case of sulfates the highest values were reached in the second irrigation application. This circumstance support the fact that the organic sulfur may have undergone organic matter mineralization processes and appeared in the leachates most significantly in the third sampling.

3.2.5 Cations

From the environmental point of view, the concentrations of Ca, Mg, Na and K in the leachates pose no risk (Nogués et al., 2000). The concentration of soluble K in the sludge does not appear to produce an increase of this element in the leachates. It is possible that the clayey nature of this substratum limits the displacement and loss of this nutrient, which can be relatively adsorbed by the clay minerals (Pond et al., 2005). For Na, a clear concentration increase was noticed in the first and second irrigation with the treatment that was not significant for the remaining samplings. The concentration of Ca seems to increase with the treatment and the sludge dose applied. Over time, its tendency is to decrease. The soil reaction with the sludge appears to have increased the concentration of soluble Ca, as it appeared in the leachates in considerable concentrations. The Mg increased significantly with the treatment, and diminished over time.

3.3 Correlations

The electrical conductivity correlated well with the concentrations of the cations analyzed in the leachates, with the exception of K^+ (Table 6). In the case of Ca, this correlation was excellent. It is obvious that the Ca in the leachates comes from both the substratum used and the sludge applied as organic amendment.

Electrical conductivity correlations with the anions concentrations presented a heterogeneous behavior. This correlation was excellent with NO_3^-, but less so for either sulfate or chloride, as it could be expected (Table 6). This may be due to greater mobility and concentration of the nitrate (Almendro-Candel et al., 2007). The resulting nitrate values in the first irrigation applications were very high and, consequently, they were washed out quickly. In the case of chlorides, the concentrations were lower.

Sulfates are salts that have a lower solubility than halite. There were significant correlations between sulfate and Mg, Ca, and K. The highest correlation was obtained with Mg^{2+} that comes mainly from the substratum formed by magnesium limestone and dolomite subjected to a crushing process in the quarry plant. This may be due to the presence of the epsomite ($MgSO_4{\cdot}7H_2O$) having a higher solubility (Jordán et al., 2004; Jordán et al., 2008) than gypsum ($CaSO_4{\cdot}2H_2O$).

The good correlations obtained between the nitrate and the alkaline earth elements and Na^+ were mainly due to their rapid percolation through the dissymmetrical columns, above all in the first irrigation (Table 7). Chlorides could replace nitrate and nitrite, and ammonium could also be exchanged by sodium, in the surface and exchange complex of the residues (Santibánez et al., 2007). After the first irrigations an equilibrium in these materials is expected and the biological activation of the media may determine the increment or descent of nitrogen in the leachates (Santibánez et al., 2007).

4 Conclusions

The good correlations obtained for some physical-chemical parameters can help to establish indicators of environmental quality of leachates over time. Therefore, constant monitoring of water quality by selecting the most appropriate indicators is recommended. As for the environmental risk with respect to the contamination of aquifers, significant nitrate concentrations appeared that may pose an environmental contamination risk. Irrigation scheduling should be an important part of a management plan in limestone quarries reclamation.

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FIGURE CAPTIONS

Figure 1. Columns used in the experiment.